

STATEMENT

As engineer responsible for the research programme on hydrophobic substrates, I have been working since October 2006 at Saint-Gobain Research on developing materials having hydrophobic properties of the type of those described in patent application EP 05728106.5 (= **instant patent application US 10/590,197**).

My functions at Saint-Gobain Research consist more particularly in seeking novel substrates having a hydrophobic functionality, i.e. those having not only improved hydrophobicity properties but also improved abrasion resistance properties as measured, for example, by the Opel or Taber tests described in patent application EP 05728106.5. In this regard, I worked on the compositions described in patent application EP 05728106.5. The work carried out by me has indicated many times to me that an ionized oxygen plasma treatment as described in patent application EP 0 476 510 A1 cannot lead to the etching of a silicon-containing layer.

With the aim of confirming this, I synthesized a substrate according to Example 2 of patent application EP 0 476 510 A1. In the synthesized example, a dense SiO₂ sublayer comparable to that described in the example was firstly formed on a glass substrate. In accordance with the example, the substrate provided with its sublayer was subjected to an activation treatment by a CVD (PE-CVD) plasma device of the type described in the prior document.

The conditions adopted were the severest possible, within the limits of our apparatus, in order to attempt to etch the oxygen-containing sublayer:

- plasma: exclusively oxygen;
- pure oxygen flow rate: 200 sccm at a pressure of 100 µbar (i.e. 75 mtorr);
- apparatus power: 230W;
- radio frequency: 13.56 MHz;
- time: 20 min.

Despite these severe conditions and the treatment time being extended to 20 minutes, no etching of the surface of the SiO₂ sublayer was observed. In particular, its RMS roughness, as measured by AFM (Atomic Force Microscope), remained unchanged.

More precisely, the roughness measurements were carried out on two regions of the substrate. The data, measured before and after the treatment, are given in Table 2:

Substrate region	RMS roughness before the treatment	RMS roughness after the treatment	Roughness R_a before the treatment	Roughness R_a after the treatment
1	1.083 nm	1.046 nm	0.882 nm	0.850 nm
2	0.984 nm	0.826 nm	0.808 nm	0.666 nm

Table 1 shows that the plasma treatment by ionized oxygen ions does not etch the layer, which therefore confirms all my previous observations. Visual inspection of the photographs (appended herewith) obtained by the AFM technique also indicate that the surface appearance of the silicon-oxide sublayers is unchanged after the plasma treatment.

In contrast, as indicated in patent application EP 05728106.5, the various trials performed at Saint-Gobain Recherche on this subject, especially under my responsibility, demonstrated that it is possible to etch the surface of the SiO_2 sublayer, but using a plasma of a fluorinated gas, optionally mixed with oxygen when a fluorocarbon gas of the CF_4 or C_2F_6 type is used. Such a process results, in my experience, to an improvement in the mechanical properties, i.e. the abrasion resistance as measured by the various tests (Taber, Opel, etc.) carried out by me, of the hydrophobic coatings finally obtained after deposition on the etched surface of the silicon-containing sublayer. This improvement was observed in particular in comparison with another activation treatment not going as far as etching, as indicated in patent application EP 05728106.5 (Example 1, Table 3).

I certify that all the statements made above are, to the extent of my current knowledge, true and result from experiments carried out either by myself or under my direction.

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